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EFFECTS OF HIGH-ENERGY IONS ON SYNTHETIC
QUARTZ

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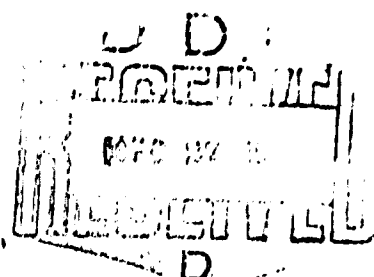
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13. ABSTRACT <p>Synthetic quartz was bombarded with 3 MeV N^+ ions at doses ranging from 4×10^{13} to 2×10^{16} N^+/cm^2 and was examined by transmission electron microscopy. The nature of the damage in unannealed and annealed specimens was characterized. In particular, it was found that increased Dauphiné inversion twinning occurred in specimens receiving doses up to $\sim 1 \times 10^{14} N^+/cm^2$.</p> <p>Reproduced by NATIONAL TECHNICAL INFORMATION SERVICE U.S. Department of Commerce Springfield VA 22151</p> <p>KEYWORDS: Quartz, Ion implantation, Electron microscopy, Radiation damage</p>
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Using a Van De Graaff Accelerator thinned specimens were subjected to bombardment by 3 MeV N^+ ions to fluences ranging from 4×10^{13} to 2×10^{16} ions/cm². They were then examined by transmission electron microscopy and reflection electron diffraction using a 100 KV electron beam.

At the lowest fluence of 4×10^{13} ions/cm² diffraction patterns of the specimens contained Kikuchi lines which appeared somewhat broader and more diffuse than those obtained on unirradiated material. No damage could be detected by transmission electron microscopy in unannealed specimens. However, Dauphiné twinning was particularly pronounced after heating to 665°C for one hour and cooling to room temperature. The twins, seen in Fig. 1, were often less than .25 μ m in size, smaller than those formed in unirradiated material and present in greater number. The results are in agreement with earlier observations on the effect of electron beam damage on Dauphiné twinning (2). Also visible are a few dislocation loops possibly due to annealing of defects caused by the ions. These loops can readily be distinguished from the black damage centers formed by the electron beam during examination of the specimen. This was discussed in a paper presented at last year's meeting of this Society (1).

At a fluence of 1×10^{14} ions/cm² Kikuchi lines were absent and extra spots appeared in the diffraction patterns. Reflection electron diffraction showed the presence of an extra phase which has not yet been identified. Transmission electron microscopy shows a heavy population of defect clusters, seen in Fig. 2, often aligned along a $\langle 10\bar{1}0 \rangle$ direction. Specimens annealed to 578°C showed little change in the clusters. The electron diffraction patterns, however, contained rings or arcs suggesting that recrystallization of amorphous material may have occurred. At 655°C the diffraction pattern indicates that the material is again mainly single crystal and the image contains many small regions of different contrast believed to represent Dauphiné twins. The small size of these twins, about 500 Å or less, can be seen in Fig. 3. Upon heating the specimen to 760°C dislocation loops and tangles of dislocations were observed.

Specimens bombarded to a fluence of 4×10^{14} ion/cm² were amorphous in the regions examined. Some recrystallization occurred at 710°C, and at 760°C the specimen was polycrystalline in all regions transparent to the beam. No crystallization occurred in specimens bombarded to fluences of 2×10^{16} ion/cm² on heating to 760°C.

Results appear to confirm earlier observations regarding the increased number of Dauphiné twins per unit area occurring in damaged regions of quartz upon annealing and cooling through the α - β transformation temperature of 574°C and suggest a correlation between fluence and size and number of twins observed. Differences in the ease of recrystallization of amorphous material may be related to the amount of crystalline material remaining after ion bombardment. For example, the failure of the specimen subjected to the highest fluence of 2×10^{16} ions/cm² to recrystallize may be attributed to a lack of crystalline nucleation sites.

1. Joseph J. Comer, 29th Annual Proceedings Electron Microscopy Society of America, Ed. C. Arceneaux (Claitor's, Baton Rouge, 1971), p.156.
2. Joseph J. Comer, to be published in J. Crystal Growth.



Fig. 1 4×10^{13} N^+ Ions/cm². Dauphine twins and dislocation loops (arrows) formed on heating to 655°C.

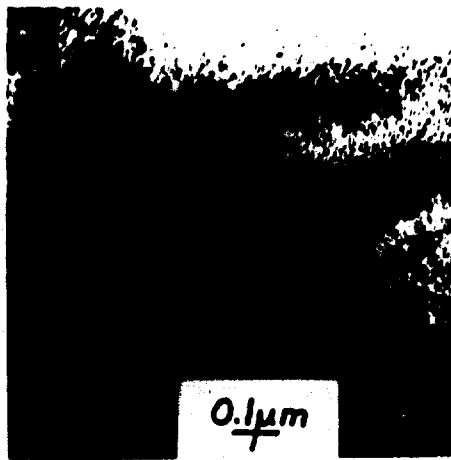


Fig. 2 1×10^{14} N^+ Ions/cm². Strain fields from defect clusters in unannealed specimen.

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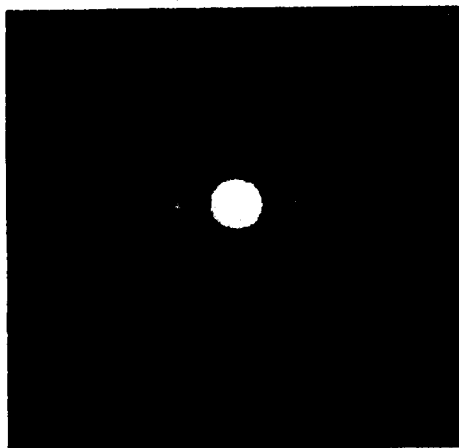


Fig. 3 1×10^{14} N^+ Ions/cm². Diffraction pattern shows some randomly oriented quartz due to recrystallization of amorphous material at 578°C.



Fig. 4 1×10^{14} N^+ Ions/cm². Annealed to 655°C. Small polygonal forms in different contrast may be Dauphine twins, regrown material or both.